WJEC (Eduqas) Chemistry A-level

SP OA4b - Synthesis of an Organic Solid Product

Methods and images taken from the WJEC practical handbook

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Aim

To **synthesise** 2-acetoxybenzenecarboxylic acid (aspirin) from 2-hydroxybenzenecarboxylic acid and ethanoic anhydride, **purify** the product using **recrystallisation** and determine its **melting point.**

Apparatus and Chemicals

- Deionised water
- Access to 3 decimal place mass balance (minimum 2 decimal place)
- 25 cm^3 pear shaped flask
- 10 cm^3 measuring cylinder
- Hot water bath
- Ice bath
- Glass stirring rod
- Buchner funnel
- Suction apparatus
- Watch glass
- Sample vial
- Labels/suitable pen
- Filter paper
- Melting point apparatus / Thiele tube
- Capillary tube
- 2-hydroxybenzenecarboxylic acid
- $(CH_3CO)_2O$ (ethanoic anhydride)
- Concentrated H_3PO_4 solution

Safety Considerations

★ 2-hydroxybenzenecarboxylic acid - harmful
★ $(CH_3CO)_2O$ - flammable, corrosive
★ Concentrated H_3PO_4 solution - corrosive

Method

1. Weigh out 1.0 g of 2-hydroxybenzenecarboxylic acid and transfer to a **pear shaped flask**.
2. Add 2 cm^3 of $(CH_3CO)_2O$ and 8 drops of concentrated H_3PO_4 solution.
3. Connect the **reflux condenser** and place in a **fume hood**.
4. Warm the mixture in a hot water bath until all of the solid dissolves then warm for a further 5 minutes.
5. Carefully add 5 cm^3 of cold deionised water to the solution and stand the flask in a bath of iced water until **precipitation** appears to be complete. It may be necessary to **stir vigorously** with a glass rod to start the precipitation process.
6. Filter the mixture under **reduced pressure** to obtain the impure derivative and wash with a little cold water.
7. Using a boiling tube in a water bath, dissolve the **impure product** in the minimum amount of warm ethanol.
8. Add 5 cm^3 of warm water drop-wise. If the solution becomes cloudy, heat until it becomes clear again.
9. Place the boiling tube into ice water for 15 minutes or until the crystals stop forming.
10. Filter the purified derivative under **reduced pressure**.
11. Dry the purified product using filter paper.
12. Collect your sample in a dry, pre-weighed sample vial and calculate the mass of the product. Use this information to calculate the **percentage yield** of the product.
13. Measure the **melting point** of the product.